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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

First Named Inventor: THOMPSON, DELTON R. JR.
Application No.: 10/728555 Confirmation No.: 9972
Filed: December 5, 2003 Group Art Unit 1732
Title: FIBROUS NONWOVEN WEBS

CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service as First Class Mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450 on:

Date July 16, 2007

Signature Judy L. Hansen
Judy L. Hansen

SECOND AMENDED BRIEF ON APPEAL

Mail Stop: Appeal Brief-Patents
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

This is an appeal from the Office Action mailed on September 1, 2006, in light of the Advisory Action mailed December 18, 2006, finally rejecting claims 1-10.

The fee has already been paid with our electronic submission dated February 12, 2007.

- ☐ Please charge the fee provided in 37 CFR § 41.20(b)(2) to Deposit Account No. 13-3723. One copy of this sheet marked duplicate is also enclosed.
- ☐ Any required fee will be paid at the time of EFS-Web submission.
- ☒ If necessary, charge any required fee, or credit any overpayment to Deposit Account No. 13-3723.

A Notice of Appeal in this application was mailed on December 21, 2006 and was received in the USPTO on December 21, 2006.

REAL PARTY IN INTEREST

The real party in interest is 3M Company (formerly known as Minnesota Mining and Manufacturing Company) of St. Paul, Minnesota and its affiliate 3M Innovative Properties Company of St. Paul, Minnesota.

RELATED APPEALS AND INTERFERENCES

Appellants are unaware of any related appeals or interferences.

STATUS OF CLAIMS

Claims 1-10 are pending, all ten claims stand rejected, and appeal is taken from the rejection of all ten claims.

STATUS OF AMENDMENTS

No amendments have been filed after the final rejection.

SUMMARY OF CLAIMED SUBJECT MATTER

The claims at issue concern a new method for preparing a nonwoven meltblown fibrous web based on polyethylene terephthalate (PET) fibers. Applicants's new method advances the art by preparing a meltblown PET web having long-sought properties of strength, durability and dimensional stability. As discussed in applicants' specification, especially page 1, line 28 through page 2, line 16, conventional meltblown PET fibers lack the crystalline orientation needed for strength and dimensional stability. Applicants' invention overcomes this problem with novel method steps that achieve chain-extended crystallization in meltblown PET fibers.

There are two independent claims – claims 1 and 5.

As recited in claim 1 applicants' new method comprises (paragraphing added):

- extruding molten PET polymer at a temperature of about 295 degrees C or less into a high velocity stream of air to produce a mass of PET fibers,

[see applicants' specification, page 8, lines 4-12 for the step of extruding molten PET into a stream of air illustrated by reference to meltblowing die 10 in FIG. 1; and see applicants' specification, page 6, lines 21-22 and page 10, lines 5-8 for a discussion of the temperature of extrusion],

- the stream of air having a temperature of about 260 degrees C or less and
[see applicants' original claim 1, and also page 8, lines 10-12; paragraph bridging pages 9 and 10; page 10, lines 12-14; and most of the examples except Examples 12-17]
- a velocity of at least 100 meters per second sufficient to impart chain-extended crystallization to the PET fibers; and
[page 6, lines 23-24 and page 10, lines 21-22 in respect to velocity; and original claim 1 and paragraph bridging pages 8 and 9, especially, page 9, lines 12-16 in respect to chain-extended crystallization]
- collecting the prepared PET fibers.
[page 8, lines 15-16]

As recited in independent claim 5, the method comprises

- heating PET polymer resin having an intrinsic viscosity of between about 0.45 and 0.6 to a molten form,
[original claim 5; see also, page 6, lines 24-25 and page 11, lines 15-16]
- extruding the molten PET polymer while at a temperature of about 285 degrees C or less through the orifices of a meltblowing die into a high-velocity stream of air to thereby prepare a mass of meltblown PET fibers having an average diameter of about 20 micrometers or less,
[see applicants' specification, page 10, line 8 in respect to the temperature of extrusion; page 8, lines 4-12 for the step of extruding molten PET into a stream of air, illustrated by reference to meltblowing die 10 in FIG. 1; and page 5, lines 15-17 for the size of the fibers]
- the stream of air having a temperature of less than about 270 degrees C and
[original claim 5 and page 10, lines 13-14]
- an air velocity of at least 100 meters per second sufficient to impart chain-extended crystallization to the PET fibers;
[page 6, lines 23-24 and page 10, lines 21-22 in respect to velocity; and original claim 5 and paragraph bridging pages 8 and 9, especially, page 9, lines 12-16 in respect to chain-extended crystallization]
- collecting the prepared PET fibers as a web; and

[page 8, lines 15-16]

- passing the collected web through an oven to anneal and autogenously bond the PET fibers together at points of fiber intersection.

[original claim 5 and page 8, lines 20-29; see FIGS. 10a and 10b and the accompanying description in the paragraph bridging pages 12 and 13 for a bond site 70]

GROUND OF REJECTION TO BE REVIEWED ON APPEAL

First Ground of Rejection

Claims 1-3 stand rejected under 35 USC § 102(b) as anticipated by Buntin et al., U.S. Patent No. 3,849,241 (hereafter Buntin).

Second Ground of Rejection

Claims 1-10 stand rejected under 35 USC § 103(a) as unpatentable over various combinations of Buntin and one or more of Thompson et al., U.S. Patent No. 5,841,081 (Thompson '081), Thompson et al., U.S. Patent No. 5,958,322 (Thompson '322), and alleged admitted prior art from Application No. 09/716,790, Paper No. 7, December 12, 2002.

ARGUMENT

I. Rejection of claims 1-3 under 35 USC § 102(b) as anticipated by Buntin et al., U.S. Patent No. 3,849,241 (hereafter Buntin)

A. The rejection is based upon an incorrect reading of Buntin (Buntin et al., U.S. Patent No. 3,849,241)

1. The rejection asserts a teaching in Buntin that is not there

The examiner's misreading of Buntin extends to several teachings, but to provide a focus to the discussion, this brief will concentrate first on the mistaken contention by the examiner that Buntin "*teaches making melt blown non-woven webs by extruding PET at a temperature of 550 F (288 C)*" (see the Final Office Action, mailed September 1, 2006, paragraph bridging pages 2 and 3; emphasis added).

The quoted statement does not appear in Buntin, and the examiner does not explain where Buntin supports such a statement. After making the quoted statement the examiner cites portions of Buntin by page and line number without discussion (Final Office Action mailed September 1, 2006, page 3, lines 2-4), but none of these identified portions contains such a statement.

Presumably the examiner's position is derived by combining two paragraphs in Buntin, one called herein the "temperature-range" passage and found at column 3, lines 36-46, and a second, called herein the "polymer-list" passage, found in column 4, lines 34-42. But neither of these paragraphs, nor any other disclosure in Buntin, supports the quoted statement, as will be seen from the following discussion.

2. What Buntin actually teaches

Buntin, an early and long-obsolete patent about melt-blowing polymeric fibers, teaches that before melt-blowing a high-viscosity thermoplastic polymer, the polymer can be subjected to a "critically controlled degradation" until the polymer has a reduced viscosity (Buntin, column 2, lines 36-58); this step is said to allow the preparation of melt-blown webs that are "substantially completely free of shot" (column 2, line 59 through column 3, line 18). The controlled degradation is accomplished by heating the

polymer, optionally in the presence of a free radical source compound, to elevated temperatures said to cause such degradation.

The cited “temperature-range” passage and “polymer-list” passage of Buntin can be summarized as follows:

The “temperature-range” passage

The “temperature-range” passage, column 3, lines 36-46, is directed to the temperature at which a polymer being treated is heated to degrade it. The key part of the passage is quoted below, in a subdivided form to emphasize the different parts of the teaching:

- i) Temperatures well above the melting point of the polymer are employed.
- ii) In the absence of free radical source compounds ... the high intrinsic viscosity resin suitably is subjected to a temperature within the range of from about 550° F. [about 288 ° C] to about 900° F., preferably from about 600° F. to about 750° F.,
- iii) for a period of time effective to cause the requisite extent of polymer degradation

The “polymer-list” passage

At column 4, lines 34-42, one column after the temperature-range passage, Buntin describes the polymers that may be used in his invention, stating:

The degraded fiber-forming thermoplastic resin ... is produced ... from thermoplastic polymer resins that are degradable ..., including polyamides, e.g., ... ; polyesters, e.g., poly(methymethacrylate) and poly(ethyleneterephthalate); polyvinyls, e.g., ... ; C3-C8 polyolefins, high density polyethylene, and mixtures thereof.

The one-word mention of polyethylene terephthalate (PET) in the just-quoted disclosure is the only mention of PET in the whole 26-column patent.

3. Buntin does not teach what the examiner contends it teaches

a. The examiner's position goes beyond the statements of the cited paragraphs -- "within" does not mean "throughout"

The precise statement in Buntin presumably relied upon by the examiner is that polymers are to be treated at "a temperature within the range from about 550° F. to about 900° F." (column 3, lines 40-41; emphasis added). Applicants read this statement by Buntin as meaning that somewhere "within" the range 550°F to about 900°F, there is "a" particular temperature or temperatures at which a particular polymer can be treated.

But the examiner expands Buntin's statement by contending that Buntin's statement means that PET is to be treated and degraded at 550° F. The examiner in effect contends that Buntin means that PET (and all other listed polymers) can be treated at all temperatures "from about 550° F. to about 900° F." How else can the examiner identify 550°F in the above-quoted statement as the temperature for treatment of PET? The only basis for the examiner's choice is that 550°F lies within the range from about 550°F to about 900°F. On that same basis the examiner could just as well choose 700°F or 800°F or 900°F.

The examiner's contention is an enlargement beyond the actual statements in Buntin. Buntin is not saying that PET can be treated at any and all temperatures within the stated range. Specifically, Buntin is not saying that PET can be treated at 550°F, as contended by the examiner.

b. Buntin's temperature range is a first condition, not a complete prescription

Buntin's teaching that treatment is to be within a temperature range from about 550°F to about 900°F is only a first condition to be satisfied. The exact temperature for any polymer depends on further conditions.

One such further condition, according to Buntin, is that the treatment temperature must be "well above the melting point" of the treated polymer (column 3, lines 36-37). In other words, the temperature of treatment must meet at least two conditions – it must be within Buntin's stated temperature range, AND it must be "well above the melting point."

By itself, Buntin's identification of the temperature range "from about 550°F to about 900°F" is incomplete and is only the first step in a teaching about treatment temperatures. Buntin's identification of a broad temperature range does not purport to

describe a specific temperature for treating a specific polymer. Further information – e.g., determining through experiment which temperature is sufficiently “well above the melting point” -- must be developed to know at what temperature the polymer is to be treated. The examiner’s contention that Buntin says to extrude PET at 550°F (288°C) is an expansion beyond what Buntin actually says, and a misreading of Buntin. This is a first reason why the rejections appealed from are incorrect.

B. Buntin lacks the enabling teaching about PET necessary to support the appealed rejections

1. The one-word mention of PET in a list of polymers does not enable practice of Buntin’s invention with PET

Buntin is really a patent about polyolefins, especially polypropylene. Polypropylene or other related polyolefins are discussed in every column of Buntin beginning with column 7. All the working examples in Buntin are directed to polypropylene or, in Examples 20 and 21, two related polyolefins.

Other than the one-word mention of PET in column 4, line 40, Buntin has no teaching addressed to PET. Among the teachings that would be necessary in Buntin to provide an enabling teaching of degrading and meltblowing PET are:

- Actual demonstration of degrading PET. Does the chemically complex, phenol-containing, ester molecule in PET actually degrade to a lower viscosity form in the same way as the rather uniform hydrocarbon molecule of polypropylene degrades? It is notoriously well known that polymers of different chemistry have a different susceptibility to degradation. And in Buntin the degradation must occur in a specific way – leaving the polymer with a reduced viscosity but a continued ability to be melt-blown into useful fibers.
- Specific discussion and exemplification of the temperatures to which PET would be heated to degrade it. Given the unique chemistry of PET, it is certain that conditions tailored specifically to PET would be required to cause the appropriate degradation, assuming such degradation is possible.
- Discussion and exemplification of the length of time required to heat PET to degrade it.

- Discussion and exemplification of a free radical source effective with PET.

None of these teachings are present. There is no demonstration that PET actually is useful in the disclosed invention, and given the significant chemical differences between polypropylene and PET, the utility of PET is a serious question. In any event, there is no description of the conditions needed to practice the invention with PET. As to PET, Buntin is simply an invitation to experiment, attempting to find conditions at which PET might degrade into a useful lower viscosity form suitable for meltblowing.

2. Buntin also does not enable applicants' claimed invention

Applicants' claimed invention has an objective that is radically different from Buntin's and does not involve Buntin's application of heat for a length of time sufficient to degrade the polymer. As discussed below in Section E, applicants' claims call for steps intended to "impart chain-extended crystallization." Applicants use low temperatures, the opposite from Buntin's high temperatures intended to degrade the polymer.

A valid rejection of claims under 35 U.S.C. 102 or 103 requires that the prior art reference on which the rejection is based have an enabling teaching as to the asserted subject matter (MPEP 2121). Buntin has no teaching of applicants' invention, and no enabling teaching about PET, the subject of applicants' invention.

C. The examiner disregards crucial facts that contradict the rejections

1. Pertinent prior art contradicts the examiner's erroneous reading of Buntin

Meyer et al., U. S. Patent No. 5,141,699 (copy of record), is directed to a technique for preparing melt-blown fibers, particularly in an oriented form. In the course of teaching oriented melt-blown fibers, Meyer also teaches the conventional preparation of melt-blown PET. Example 3 of the patent is a fully described process for melt-blowing PET. It teaches melt-blowing PET at temperatures of 315°C with an orienting chamber, or 335°C without an orienting chamber (the comparative example). The comparative example, without an orienting chamber, is in effect the conventional practice in melt-blowing PET.

Meyer issued in 1992, almost twenty years after Buntin, and is the only evidence of record showing conventional practice in meltblowing PET. There is no art of record demonstrating meltblowing of PET at a temperature lower than 315°C.

Meyer first of all undermines the examiner's contention that (allegedly, according to Buntin) PET will be degraded at the examiner's chosen 288°C. Both the 315°C and 335°C temperatures used in Meyer to melt-blow PET are well in excess of the 288 °C temperature selected by the examiner, and Meyer reveals no degradation occurring at those temperatures.

Meyer also confirms the novelty of applicants' invention. Both of the 315°C and 335°C temperatures used by Meyer for meltblowing PET are well in excess of the 295°C called for in applicants' claims. On the art of record, applicants' use of 295°C or less to meltblow PET is novel and unsuggested.

2. The rejection violates Buntin's requirement that the treatment temperature be "well above the melting point" of PET

As noted, Buntin states that the temperature of treatment is to be "well above the melting point" of the treated polymer" (column 3, lines 36-37). The need for high temperatures follows from the fact that the goal of the treatment is to degrade the polymer, breaking it up into a lower-viscosity form.

Buntin's working examples are the only indication in Buntin as to what is meant by "well above the melting point." Two polymers – polypropylene and poly-4-methylpentene-1 – are exemplified in Buntin's examples, and they have melting points as follows:

Polypropylene	160-170° C
Poly-4-methylpentene-1	240° C

(see the *Encyclopedia of Polymer Chemistry*, volume 8, page 26, copy attached to applicants' Response filed November 16, 2006 as Attachment N).

In Buntin's working examples polypropylene is always heated to a temperature above 580° F (about 304° C) and poly-4-methylpentene-1 is always heated above 620°F (about 326°C) for poly-4-methylpentene-1. That means the treatment temperatures for poly-4-methylpentene-1 are above the melting point of poly-4-methylpentene-1 by at least 86°C, and the treatment temperatures for polypropylene are above the melting

point of polypropylene by at least 134 to 144°C. In fact, most of Buntin's polypropylene working examples use treatment temperatures of at least 600° F (about 316°C), which is nearly 150°C above the melting point of polypropylene.

These temperature differences above melting point – 86°C to 150°C -- provide an indication as to what Buntin means by “well above the melting point.” Buntin uses high temperatures, substantially above the melting point, because polymers normally do not degrade at the melting point, and a much higher temperature is needed in order to degrade the polymer.

PET has a melting point of about 250-260°C (see Volume 8, page 26 of the *Encyclopedia of Polymer Chemistry*; Attachment N). Adding 86°C, Buntin's smallest elevation above melting point, to PET's melting point would make the treatment temperature to degrade PET at least 336-346° C. More typically, the working examples in Buntin would suggest increases above melting point of at least 150° C above the melting point, meaning a treatment temperature to degrade PET of at least 400-410° C. Whether even such temperatures would be adequate would require added information beyond any information available from Buntin, e.g., separate experimental testing.

At a minimum, the temperatures indicated by Buntin's teachings, extrapolated to PET, are far above the 295° C called for in applicants' broadest claim. On this ground alone, Buntin does not anticipate applicants' invention.

D. Applicants' claimed invention involves additional steps not taught in Buntin

The above discussion has focused on the recitation in applicants' claims calling for extruding PET polymer having a temperature of about 295°C or less; but applicants' claims also recite that the stream of air into which the polymer is extruded has a manifold air temperature of about 260°C or less. To deal with this added recitation of air temperature the examiner cites Buntin, column 7, lines 59-64, which states that the air temperature of the air stream into which the polymer is extruded “may vary from 500°F to 900°F.” The recited 500°F is equal to 260°C, and the examiner argues that Buntin teaches “extruding PET at a temperature of 550 F (288 C) ... with a stream of air at 500 F (260 C), which reads on the claimed range of less than about 260 C given the range implied by ‘about’ ” (Final Office Action, paragraph bridging pages 2 and 3).

In other words, the examiner takes the bottom temperature of the 550°F to 900°F range for treatment temperature and combines that with the bottom temperature of the 500°F to 900°F range for air temperature. Just as there is no basis for taking the 550°F temperature for PET, as discussed above, there is also no basis for taking the 500°F air temperature. Even more remote is any basis for taking the combination of the lowest treatment temperature with the lowest air temperature.

Buntin's teachings about air temperature also lead away from applicants' invention. Buntin states that the air temperature is "usually ... slightly higher, about 50°F., than nozzle die temperatures" (Buntin, column 7, lines 62-64). Since Buntin states that the nozzle temperatures are in the range from about 550°F to 900°F (Buntin, column 9, lines 16-18), Buntin's statement implies an air temperature of 600°F, which leads away from 500°F for air temperature.

More than that, there again is no basis for choosing any particular temperature for PET. Buntin's teachings about PET are so incomplete as to make the examiner's choices arbitrary. The examiner's temperature choices use applicants' claims as a guide rather than any internal teaching in Buntin leading to the combination of an extrusion temperature of 288°C and an air temperature of 260°C.

E. Buntin's method of *degrading* does not anticipate or suggest applicants' method of *meltblowing*

Buntin's temperature range "from about 550° F. to about 900° F." relied on in the rejection is part of a teaching about degrading a polymer, a separate step that Buntin uses before the step of meltblowing the polymer. The whole paragraph in Buntin in which the temperature range appears is directed to "approaches to bring about the extent of polymer degradation requisite to practice of this invention" (column 3, lines 34-36). To achieve degradation the polymer resin being degraded "is subjected to a temperature within the range from about 550° F. to about 900° F. ... for a period of time effective to cause the requisite extent of resin degradation" (column 3, lines 40-44). The temperature range is thus chosen for its purpose of causing polymer degradation, not polymer meltblowing.

Applicants' purpose is completely different from Buntin's purpose. Applicants' purpose is to prepare meltblown PET fibers with a more fully developed molecular structure, that is, with a chain-extended crystal structure. To do that, applicants teach use of a *low* extrusion temperature. As stated in applicants' specification, page 10,

lines 5-14, the temperature of the polymer in the die cavity (from which it is extruded) “is held to a temperature less than about 35°C higher than the melting point ... [and] preferably ... no more than about 20°C higher than the melting point” (see applicants’ specification, page 10, lines 5-14). Also, applicants extrude the polymer into an air stream that has a low temperature.

Applicants advanced the art by achieving a dimensionally stable nonwoven web of PET meltblown fibers. Others had tried different approaches to dimensionally stabilize meltblown webs of PET fibers, but no successful product had been obtained; see applicants’ specification, page 2, line 17, to page 3, line 21. Applicants achieved this result directly from the meltblowing process, with no intervening or additional steps. The fact that many efforts had been made to achieve such a result is evidence that applicants’ invention was not obvious.

Buntin has absolutely nothing to do with preparing dimensionally stable PET fibers, and Buntin offers not a hint leading to the successful preparation of such fibers and webs made from such fibers. Applicants have made a major advance in the art and Buntin in no way leads to that advance.

II Rejections of claims 1-10 under 35 USC § 103

On pages 3-12 of the Final Office Action mailed September 1, 2006 the examiner states various rejections of claims 1-10 as unpatentable over various combinations of alleged prior art under 35 USC § 103. But all of these rejections depend on the same contention by the examiner as discussed above in Section I of this brief, namely a contention that Buntin “*teaches making melt blown non-woven webs by extruding PET at a temperature of 550 F (288 C).*” For the examiner’s reliance on this asserted teaching in all of the rejections under 35 USC § 103, see the Final Office Action, page 4, first paragraph (for the rejection of applicants’ claim 4); paragraph bridging pages 4 and 5 (claim 5); third full paragraph on page 6 (claims 8 and 9); second and third full paragraphs on page 7 (further rejection of claims 1-3); page 8, last paragraph (further rejection of claim 4); paragraph bridging pages 9 and 10 (further rejection of claims 5-7 and 10); and paragraph bridging pages 11 and 12 (further rejection of claims 8 and 9).

As discussed above Buntin does not make the quoted teaching asserted by the examiner. Because the examiner's assertion is incorrect, and because each and all of the rejections depend on this assertion, all the rejections are incorrect and should be withdrawn.

CONCLUSION

For the foregoing reasons, appellants respectfully submit that the rejections stated in the Final Office Action of September 1, 2006, maintained in the Advisory Action mailed December 18, 2006, are in error and should be reversed on all counts.

Respectfully submitted,

July 16, 2007
Date

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#335039 - 56109US011 Second Amended Brief on Appeal
Office of Intellectual Property Counsel
3M Innovative Properties Company
Facsimile No.: 651-736-3833

CLAIMS APPENDIX

1. A method for preparing a nonwoven meltblown PET-fiber-based web comprising a) extruding molten PET polymer having a temperature of about 295 degrees C or less through the orifices of a meltblowing die into a high-velocity stream of air to produce a mass of PET fibers, the stream of air having a manifold air temperature of about 260 degrees C or less and an air velocity of at least 100 meters per second sufficient to impart chain-extended crystallization to the PET fibers; and b) collecting the prepared PET fibers.

2. A method of claim 1 in which the PET fibers are prepared from resin exhibiting an intrinsic viscosity of between about 0.45 and 0.75.

3. A method of claim 1 in which the prepared PET fibers exhibit a double melting peak on a DSC plot which is representative of a first molecular portion within the fiber that comprises a non-chain-extended crystalline phase, and a second molecular portion within the fiber that comprises a chain-extended crystalline phase and melts at an elevated temperature over that of the non-chain-extended crystalline phase.

4. A method of claim 1 in which other fibers or particles are dispersed among the PET fibers before they are collected.

5. A method for preparing a nonwoven meltblown PET-fiber-based web comprising a) heating PET polymer resin having an intrinsic viscosity of between about 0.45 and 0.6 to a molten form, extruding the molten PET polymer while at a temperature of about 285 degrees C or less through the orifices of a meltblowing die into a high-velocity stream of air to thereby prepare a mass of meltblown PET fibers having an average diameter of about 20 micrometers or less, the stream of air having a temperature of less than about 270 degrees C and an air velocity of at least 100 meters per second sufficient to impart chain-extended crystallization to the PET fibers; b) collecting the prepared PET fibers as a web; and c) passing the collected web through an oven to anneal and autogenously bond the PET fibers together at points of fiber intersection.

6. A method of claim 5 in which the PET polymer has a temperature of about 275 degrees C or less when extruded through the orifices of the meltblowing die.

7. A method of claim 5 in which the stream of air has an air velocity of at least 150 meters per second.

8. A method of claim 5 which includes the further step of introducing additional fibers into the stream of prepared PET fibers before collecting the web of fibers.

9. A method of claim 8 in which the additional fibers comprise staple fibers.

10. A method of claim 5 in which at least one additional polymeric material is extruded through the orifices of the meltblowing die together with the PET polymer to thereby prepare bicomponent fibers.

EVIDENCE APPENDIX

In a Response Under 37 CFR §1.116, dated November 16, 2006, applicants submitted seven pages copied from the *Encyclopedia of Polymer Science and Engineering*, Volumes 4 and 8. Four pages were from Volume 4 and were labeled Attachment M and three pages were from Volume 8 and were labeled Attachment N. Although the examiner did not formally enter this evidence he did refer to it and use it, for example, in an Advisory Action mailed December 18, 2006, page 3, seventh paragraph on the page.

Copies of the seven pages from the *Encyclopedia of Polymer Science and Engineering* submitted by applicants are attached.

RELATED PROCEEDINGS APPENDIX

None.

Attachment M

7/10/92

ENCYCLOPEDIA OF POLYMER SCIENCE AND ENGINEERING

VOLUME 4

Composites, Fabrication
to
Die Design

A WILEY-INTERSCIENCE PUBLICATION

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Table 10. Susceptibility of Unstabilized Polymers to Degradation

Polymer	Resistance to degradative process ^a				
	Thermal oxidation	Photo-oxidation, weathering	Ozone	Hydrolysis	γ -Oxidation
polyethylene	p	p	e	e	f
polypropylene	vp	vp	e	e	p
polystyrene	f	p	e	e	g
poly(methyl methacrylate)	g	e	e	g	f
poly(tetrafluoroethylene)	e	e	e	e	vp
polyamide ^b	f	f	f	f	f
polyacrylonitrile	p	g	e	g	f
poly(vinyl chloride)	vp	p	e	e	p
poly(ethylene terephthalate)	g	g	e	g	g
polyoxymethylene	p	p	f	f	vp
polycarbonate	f	p	g	g	g
poly(phenylene oxide)	vp	f	g	g	g
poly(ester urethane)	f	f	g	g	f
poly(ether urethane)	p	p	g	f	f
poly(<i>m</i> -phenylene isophthalamide)	e	vp	e	g	e
poly(<i>p</i> -phenylene terephthalamide)	e	vp	e	g	e

^a Key: e = excellent, g = good, f = fair, p = poor, and vp = very poor.

^b Nylon-6 and -6,6.

elevated temperature index values. These values are derived by Underwriters Laboratories and are recommended, maximum-use temperatures at which a 50% decrease in properties occurs in 11,000 h of exposure. These temperatures are listed in Table 11 and may be regarded as upper limits for completely stabilizer-free polymers because the precise composition is not given. The test parameters

Table 11. Thermal Deterioration of Polymers: Maximum Use Temperatures^a

Polymer generic type	Film thickness, mm	Maximum use temperature, °C
poly(vinyl chloride)	2.0	50
polyethylene	1.5	50
polyoxymethylene	0.7	50
poly(methyl methacrylate)	1.5	50
polystyrene	3.1	50
poly(phenylene oxide)	1.5	50
polyamide	0.7	65
polycarbonate ^b	0.7	65
epoxy resins	3.0	90
silicones	0.7	105
poly(ethylene terephthalate)	0.2	105
alkyd resin	1.5	130
polytetrafluoroethylene	0.9	150
phenolic resin	2.0	150
polyimide	0.1	200
aramid ^c	0.2	220

^a Recommended temperature at which 50% of the original dielectric strength, tensile, and impact properties are retained for 11,000 h under low continuous stress. In some cases, stabilizers may be present. Underwriters Laboratories data (83).

^b Bisphenol A.

^c Aromatic polyamide.

employed include dielectric strength, tensile strength, and impact resistance, but all give very similar temperature indexes. A more complete listing of plastics may be found in Ref. 83; data tables also show the changes in tensile strength of many types of plastics upon exposure to acids and bases (see CHEMICALLY RESISTANT POLYMERS and articles on individual polymers).

The cross-correlation of the various physical and chemical changes that result from a given degree of degradation for a specific polymer is important. However, there are relatively few comprehensive studies on well-characterized, unstabilized samples, and many studies rely on the measurement of a single parameter.

Polyolefins

Polyolefins, most notably PE and PP, find application in many areas where degradation is a serious problem. For example, large quantities are used in film, as dielectric or insulating layers and coatings, and in injection or blow molding, especially for automotive and appliance components and for pipe (qv). PP fibers are widely used in both domestic and industrial applications. Polyolefins are prone to degradation by thermo- and photooxidative processes, as well as the less common effects of γ -radiation and potent acidic media. In all uses, the rate of attack on PP is greater than on PE; differences between LDPE and HDPE are clouded by both chemical effects, ie, frequency of tertiary C—H sites, and physical effects. HDPE crystallinity is usually much greater than for LDPE (see also ETHYLENE POLYMERS; PROPYLENE POLYMERS).

Thermal Oxidation. Following the initial thermal generation of free radicals, polyolefin oxidation is believed to follow equations 22 to 26. The changes occurring in PE under simulated injection-molding conditions with varying, low amounts of O_2 have been explored (60). At very low O_2 concentration, ie, $\leq 5 \times 10^{-4}\%$, at $\leq 315^\circ\text{C}$, the molecular weight distribution of LDPE shifts to higher values, whereas at $\geq 330^\circ\text{C}$ a lowering is observed. Changes in long-chain branching are also observed. At ca 1.2% O_2 in LDPE, chain scission and long-chain branching occur simultaneously at ca 300°C . The presence of TiO_2 pigment can affect the thermal degradation of LDPE (50). Uncoated rutile promotes degradation, whereas coated pigment particles slow the drop in molecular weight.

The generation of peroxides has been correlated with changes in molecular weight distribution for LDPE during melt processing at 150 – 175°C in a torque rheometer (65). Unstabilized PP also undergoes degradation during processing as the result of thermal and mechanical-scission processes (65). The presence of O_2 or deliberately added peroxides at extrusion temperatures, ie, 180 – 250°C , leads to a very rapid drop in molecular weight by random scission processes. These processes are used to adjust the molecular weight distribution of PP, so that a polymer may be optimized for different uses.

Several attempts have been made to characterize the oxidation products from polyolefins under various thermal, photo-, and γ -initiation conditions. Some of these data are collected in Table 12 for comparative purposes. In one of the most comprehensive approaches (78), nmr and O_2 -uptake data give a detailed picture of the melt oxidation products from LDPE (Fig. 3). The nmr technique suffers from low sensitivity; the data at 12 mL O_2 /g corresponds to ca 0.5 mol oxidation/kg LDPE, but the excellent resolution of the technique allows identi-

Attachment IV

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Identification
to
Lignin

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ranges that are usually less sharply defined than the melting points of low molecular-weight crystalline materials. Above the flow temperature, the sample begins to break down chemically (pyrolysis). This process of thermal degradation produces low molecular-weight fragments, which are often flammable or have a characteristic odor.

In some cases, softening or melting occurs above the range in which the polymers are thermally stable, i.e., decomposition starts before melting of the sample can be observed. In cross-linked plastics there is usually no softening until just below the point of chemical degradation. Therefore, this behavior is an indication, although not unequivocal, that the material is a cured thermoset. Thermosets and elastomers show little or no flow up to their decomposition temperature, where they form typical degradation products, characteristic of the particular plastic.

The softening range of a plastic can be determined by the usual methods of organic chemistry, either in a melting-point tube or with a hot-stage microscope. However, the resulting values often depend on the rate of heating and on the presence of additives, especially plasticizers. Values for the most important plastics are shown in Table 3 and more comprehensive tabulation is available (1).

Table 3. Softening and Melting Ranges of Thermoplastics

Thermoplastic	Softening or melting range, °C
poly(vinyl acetate)	35-85
polystyrene	70-115
poly(vinyl chloride)	75-90 (softens)
polyethylene, density 0.92 g/cm ³	~110
density 0.94 g/cm ³	~120
density 0.96 g/cm ³	~130
poly(1-butene)	125-135
poly(vinylidene chloride)	115-140 (softens)
poly(methyl methacrylate)	120-160
cellulose acetate	125-175
polyacrylonitrile	130-150 (softens)
polyoxymethylene	165-185
polypropylene	160-170
nylon-12	170-180
nylon-11	180-190
polytrifluorochloroethylene	200-220
nylon-6,10	210-220
nylon-6	215-225
polycarbonate	220-230
poly(4-methyl-1-pentene)	240
nylon-6,6	250-260
poly(ethylene terephthalate)	250-260

Pyrolysis Tests. In order to examine the behavior of a plastic on heating without direct exposure to flame, a small sample is placed in a pyrolysis tube held at the upper end with a clip or pair of tongs. A piece of moist litmus or pH paper is placed at the open end of the tube. The tube is heated slowly so that the